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<p align="center">Division of Forensic Science</p> <p align="center">TRACE EVIDENCE PROCEDURES MANUAL</p>	<p align="center">Amendment Designator:</p>
	<p align="center">Effective Date: 31-March-2003</p>
<p align="center">8 GENERAL CHEMICAL</p> <p>8.1 Analytical Approach</p> <p>8.1.1 Items submitted for general chemical examinations are the miscellaneous samples not included in another examination (e.g., greases, glues, cosmetics, unknown substances). They are often referred to as any chemistry-related exams requested of the laboratory that are <u>not</u> a controlled substance.</p> <p>8.1.2 While the Section sees some evidence types on a fairly routine basis, others may require method development at the time the case is being worked. No procedures manual could encompass methods for every general chemical evidence type analyzed. This section will provide analysis schemes for some of those that the Section has analyzed with some regularity.</p> <p>8.1.3 Good scientific principles and a logical analysis scheme are applied to those evidence types that have not been encountered before at the discretion of the examiner based upon the type and amount of evidence submitted and the case information received. Generally speaking, a visual macroscopic/ microscopic examination will be performed (stereo, compound, comparison and polarizing microscopes) for color, texture, viscosity, morphology, optical properties and homogeneity.</p> <p>8.1.4 The amount of sample will determine how the examiner proceeds. With a limited amount of sample, nondestructive testing is performed first. Solubility/miscibility tests with organic/inorganic and polar/nonpolar solvents (e.g., water, carbon disulfide, pentane, acetone, chloroform) may be done. The pH of water soluble/miscible samples may be taken using pH paper or a pH meter. Microchemical tests may be performed as indicators of a class of compounds present (e.g., for sulfates, chlorides, nitrates, sugars).</p> <p>8.1.5 Necessary instrumentation is used to identify or to compare samples.</p> <p>8.2 Minimum Standards and Controls, Documentation, Report Wording</p> <p>8.2.1 These are dependent upon the type of evidence being submitted and the examinations being performed.</p> <p>8.2.2 Generally, blanks, reference standards and controls are run along with the evidence items and all of these results are recorded in the case file notes. All documentation is included to support the conclusion made. Instrument output requirements are detailed throughout other sections of this manual. Report wording is as consistent as possible with previously reported exams of a given type. When reporting conclusions based upon the examination of “new” evidence types the supervisor and/or technical reviewer will help ensure that the report wording is accurate, clear and concise.</p> <p>8.3 Acids and Bases</p> <p>8.3.1 Purpose</p> <p>To identify common acids and bases that may be encountered in casework.</p> <p>8.3.2 Safety Considerations</p> <p>8.3.2.1 Acids and bases, e.g., HCl, HNO₃, H₂SO₄, H₃PO₄, NH₄OH, NaOH, KOH, may be encountered as evidence. These are very corrosive. Eye and skin protection must be used.</p> <p>8.3.2.2 Acids may be very reactive with chlorates, acetone, flammable liquids and water. Extreme care must be taken when mixing these compounds.</p> <p>8.3.3 Minimum Standards and Controls</p> <p>8.3.3.1 Treat the questioned samples and any control/standard samples in the same manner.</p>	

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8.3.3.2	When performing extractions also analyze a blank of the extraction liquid and an undisturbed portion of the substrate, if possible.	
8.3.3.3	Controls and blanks will be run with each case when performing testing with microchemical spot test reagents. These results will be recorded in the case file notes.	
8.3.4	Analytical Procedures	
8.3.4.1	If the sample is a solid or is on a solid substrate, dissolve in DI H ₂ O. If the sample is a liquid, check if it is miscible with H ₂ O by adding a small amount of the liquid to H ₂ O.	
8.3.4.2	A preliminary pH may be determined with pH paper. A more accurate value should be obtained from the use of a pH meter.	
8.3.4.3	If the pH is acidic proceed as follows:	
8.3.4.3.1	Hydrochloric acid: Test unknown with a few drops of silver nitrate reagent. A white precipitate indicates the presence of a chloride.	
8.3.4.3.2	Sulfuric acid: Test unknown with a few drops of barium chloride reagent. A white precipitate indicates the presence of a sulfate.	
8.3.4.3.3	Nitric acid: Test unknown with a few drops of diphenylamine reagent. Immediate development of a deep blue color indicates the presence of nitrates.	
8.3.4.3.4	Phosphoric acid: Test unknown by placing 6 ml of acetone in a small test tube. Place 3 drops of unknown liquid into test tube. Mix. Add 3 drops of ammonium hydroxide and mix. A precipitate indicates the presence of ammonium phosphate dibasic, (NH ₄) ₂ HPO ₄ , if phosphoric acid is present.	
8.3.4.3.5	Specific anion test strips (such as EM Quant [®]) may be used in place of, or in addition to, any of the above microchemical tests. While these test strips may have an expiration date they will continue to be used as long as they test positive with an appropriate known acid.	
8.3.4.4	If the pH is basic proceed as follows:	
8.3.4.4.1	Ammonium hydroxide: Test the unknown with Nessler reagent. The formation of an orange to brown precipitate indicates the presence of ammonium ions. Ammonium test strips (such as EM Quant [®]) may be used in place of, or in addition to, the Nessler reagent test.	
8.3.4.4.2	Sodium hydroxide: Basic pH and SEM/EDS.	
8.3.4.4.3	Potassium hydroxide: Basic pH and SEM/EDS.	
8.3.4.4.4	A report may be generated at this time if the results of the pH test and the spot tests are sufficient for the case report.	
8.3.4.4.5	If further characterization of the acid/base is necessary, then the extract and/or the precipitate from the microchemical testing may either be run using X-ray diffraction, ion chromatography or FTIR, each combined with SEM/EDS, as appropriate.	

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8.3.5	Report Wording	
8.3.5.1	If the pH is neither acidic nor basic report: No acids or bases were identified in Item ____ or the Item ____ extract. (As appropriate)	
8.3.5.2	If the results of the pH test and the spot tests are sufficient for the case report: The Item ____ extract was indicative of _____ (name of acid or base).	
8.3.5.3	If identifying the acid or base: Item 1 is an acidic/basic solution that contained _____ (name of acid or base).	
8.3.5.3.1	Alternatively, the following may be used as appropriate: Item 1 is an acidic/basic solution consistent with _____ (name of acid or base).	
8.3.5.3.2	If appropriate, common sources of the acid/base may be included:	
8.3.5.3.2.1	Hydrochloric acid, commonly found as muriatic acid.	
8.3.5.3.2.2	Sulfuric acid, commonly found as battery acid.	
8.3.6	References	
8.3.6.1	Anger, V., and Feigl, F., <u>Spot Tests in Inorganic Analysis</u> , 6 th ed., Elsevier Publishing Company: Amsterdam, The Netherlands, 1972.	
8.3.6.2	Illinois State Police, Trace Chemistry Procedures Manual, Common Acids Identification, February 1, 1997.	
8.3.6.3	Jungreis, Ervin.; "Spot Test Analysis, Clinical, Environmental, Forensic, and Geochemical Applications", Volume 75, 1985.	
8.4	Bank Dyes	
8.4.1	Purpose The most common exploding bank dye packs encountered in casework have contained the red dye, 1-methylaminoanthraquinone (MAAQ), and the lachrymator, o-chlorobenzylidenemalononitrile (CS). The red dye, 1-methylaminoanthraquinone, may also be found in devices containing red smoke mixtures. This procedure describes a means of extracting and identifying these components.	
8.4.2	Safety Considerations	
8.4.2.1	Care should be taken as MAAQ is a prolific dye that does not wash off with water.	
8.4.2.2	Lachrymators, such as CS, are compounds that irritate the eyes and mucous membranes and should be handled accordingly.	
8.4.3	Minimum Standards and Controls	
8.4.3.1	Treat the questioned samples and any control/standard samples in the same manner.	

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8.4.3.2	When performing extractions also analyze a blank of the extraction liquid and an undisturbed portion of the substrate, if possible.	
8.4.4	Analytical Procedures	
8.4.4.1	Visually examine the evidence for any staining of a red dye.	
8.4.4.2	Suitable areas are extracted with a minimum volume of chloroform or methanol.	
8.4.4.3	Use the same approximate size of unstained material, if available, for the substrate control.	
8.4.4.4	If the extract is relatively “clean”, the red dye may be identified using FTIR.	
8.4.4.5	Screen the extract on the GC using standard fire debris analysis conditions for peaks corresponding to both CS and MAAQ.	
8.4.4.6	If no peaks are present at the proper retention times, write a negative report.	
8.4.4.7	If peaks are present at the proper retention times for CS and/or MAAQ, analyze the extract by GC/MS.	
8.4.4.8	Both the dye and the lachrymator should be identified when possible.	
8.4.5	Report Wording	
8.4.5.1	<p>If a known bank dye pack is submitted and the dye is soluble in chloroform or methanol and none of the stains on the evidence extract into the solvent:</p> <p>The red stains on Item ____ could not be associated with the red dye in Item ____ due to differences in physical properties.</p>	
8.4.5.2	<p>If NO known bank dye pack is submitted and all of the stains on the evidence do NOT extract into chloroform or methanol:</p> <p>The red stains on Item ____ could not be associated with the red dye commonly used in exploding bank dye packs due to differences in physical properties.</p>	
8.4.5.3	If corresponding GC peaks CANNOT be identified as CS and/or MAAQ:	
8.4.5.3.1	No 1-methylaminoanthraquinone, a red dye commonly found in exploding bank dye packs, was identified in the Item ____ extract.	
8.4.5.3.2	No 1-methylaminoanthraquinone or o-chlorobenzylidenemalononitrile, common components of exploding back dye packs, was identified in the Item ____ extract.	
8.4.5.3.3	If appropriate: The extract of Item ____ could not be associated with ____ due to differences in chemical properties.	
8.4.5.4	If corresponding GC peaks CAN be identified as CS and/or MAAQ:	
	The extract of Item ____ contained 1-methylaminoanthraquinone, a red dye, and o-chlorobenzylidenemalononitrile, a lachrymator. It should be noted that these materials are commonly found in exploding bank dye packs.	

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8.4.6	References	
8.4.6.1	Martz, R.M., Reutter, D.J., and Lasswell, III, L.D., "A Comparison of Ionization Techniques for Gas Chromatography/Mass Spectroscopy Analysis of Dye and Lachrymator Residues from Exploding Bank Security Devices", <i>Journal of Forensic Sciences</i> , Vol. 28, No. 1, January 1983, pp. 200-207.	
8.4.6.2	Personal Communication, ICI Americas, Inc., August 1981.	
8.5	Salt	
8.5.1	Purpose	
	To identify sodium chloride (NaCl) and other salts that may be encountered in casework. The most routine cases requesting this exam are deer "baiting" cases but the analysis scheme is readily applied to other situations as well.	
8.5.2	Minimum Standards and Controls	
8.5.2.1	Treat the questioned samples and any submitted control/standard samples in the same manner.	
8.5.2.2	Controls and blanks will be run with each case when performing testing with microchemical spot test reagents. These results will be recorded in the case file notes.	
8.5.3	Analytical Procedures	
8.5.3.1	Examine the sample(s) with the aid of the stereomicroscope to determine if there are any obvious cubic crystals present. If cubic crystals are observed, remove them and perform identification by X-ray diffraction.	
8.5.3.2	If cubic crystals are not observed, crush the sample to a uniform mesh size and mix well.	
8.5.3.3	Weigh the samples. Use about one-half of the smallest sample's weight as the standard weight for all extraction samples.	
8.5.3.4	Place each sample in a labeled beaker and add enough D.I. water to each beaker to cover the sample. Stir and then sonicate for 10 minutes.	
8.5.3.5	Centrifuge and/or filter the samples and transfer the extract to a small beaker.	
8.5.3.6	Record the pH of the extract.	
8.5.3.7	Perform silver nitrate and barium sulfate spot tests on the extract and record the results. If no precipitate forms, the results are negative and no further examination is necessary.	
	8.5.3.7.1	If the results are indicative of a chloride, remove a portion of the extract for IC analysis. Evaporate the remaining portion to dryness and analyze the residue by X-ray diffraction and, if necessary, SEM/EDS. If necessary, the H ₂ O extract may be analyzed by IC and SEM/EDS.
8.5.3.8	Return the original samples and the extracted samples, which have been dried, properly labeled as such.	
8.5.4	Report Wording	
8.5.4.1	If testing is negative for sodium chloride:	
	No sodium chloride was identified in the Item __ extract.	

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<div> <div>8.5.4.2</div> <div>If sodium chloride crystals are present: The white crystalline material in Item ___ was identified as sodium chloride, commonly known as table salt.</div> </div> <div> <div>8.5.4.3</div> <div>If sodium chloride is identified from a water extract: The extract of Item ___ contained sodium chloride, commonly found in table salt.</div> </div> <div> <div>8.5.4.4</div> <div>If the RFLE does not contain a specific request for sodium chloride it may be appropriate to say: No salts were identified in the Item ___ extract.</div> </div>	
<p>8.6 Sugars</p>	
<div> <div>8.6.1</div> <div>Purpose To identify common sugars (such as sucrose, dextrose, fructose or lactose) that may be encountered in casework. Sugar may be encountered in the vandalism of motor vehicles by its addition to the fuel supply or the crankcase oil. Because the solubility of sugar in gasoline is so low (1.5 mg/L), a sampling of any solid residues in the fuel tank as well as the fuel or oil filters, as appropriate, should be included. Sugars may also be encountered as a cutting agent in controlled substance cases or as part of a sugar/chlorate mixture in improvised explosive devices.</div> </div>	
<div> <div>8.6.2</div> <div>Minimum Standards and Controls 8.6.2.1 Treat the questioned samples and any control/standard samples in the same manner. 8.6.2.2 When performing extractions also analyze a blank of the extraction liquid and an undisturbed portion of the substrate, if possible. 8.6.2.3 Controls and blanks will be run with each case when performing testing with microchemical spot test reagents. These results will be recorded in the case file notes.</div> </div>	
<div> <div>8.6.3</div> <div>Analytical Procedures 8.6.3.1 Stereoscopically examine the sample(s) to determine if there are any obvious crystals present and if the sample is homogeneous. If crystals are observed, remove them and perform solubility tests (aqueous/organic, such as water and chloroform, sugars are soluble in water). Use either the FTIR or the XRD to identify the material. 8.6.3.2 If solid material is present, it may be necessary to wash away residue left on the solid material (for instance, gasoline in vandalism cases). Sugar is not soluble in pentane and this, or another suitable solvent may be used. 8.6.3.3 If there is no solid material present, perform a water extraction of the sample containing the suspected sugar. (If the case involves an improvised explosive device, follow the procedures for explosives analysis.) Evaporate to dryness. 8.6.3.4 Perform either the Fehling's, triphenyltetrazolium chloride, naphthol or anthrone microchemical spot test. (With Fehling's or triphenyltetrazolium chloride, remember to acidify sucrose when performing the test. Run both sucrose and a known reducing sugar as positive controls.) A negative test is indicative of no sugars being detected.</div> </div>	

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8.6.3.4.1	Anthrone Microchemical Spot Test Place several crystals of anthrone in a white spot plate. A powder sample, or an aqueous solution of the unknown, is added and mixed well. Three drops of sulfuric acid are added drop wise; a blue to blue-green color formation indicates the presence of sugar. Notes: The color may take up to 5 minutes to form. Anthrone is normally used to detect carbohydrates. The test is most sensitive when a dry sample is ground with the anthrone prior to acid addition. Dilute water extracts are best evaporated to dryness prior to testing.	
8.6.3.5	Use either the FTIR or the XRD to identify the extracted residues.	
8.6.3.6	Alternatively, use the trimethylsilylimidazole (TMSI) in pyridine derivatization procedure from Pierce Chemical Company.	
8.6.3.7	Screen the derivative on the gas chromatograph and identify, as necessary, using the gas chromatograph/mass spectrometer.	
8.6.4	Report Wording	
8.6.4.1	If the questioned sample exhibits a negative microchemical spot test: No sugars were identified in the Item ____ extract.	
8.6.4.2	If the solid material or the extracted residues are identified as a sugar: <div> <div>8.6.4.2.1</div> <div>The Item ____ white crystalline solid was identified as _____ (name of sugar).</div> </div> <div> <div>8.6.4.2.2</div> <div>The Item ____ extract contained _____ (name of sugar).</div> </div>	
8.6.4.3	If appropriate, common sources of the sugar may be named: The Item ____ white crystalline solid was identified as sucrose, commonly referred to as table sugar.	
8.6.5	References	
8.6.5.1	Feigl, F. "Spot Tests In Organic Analysis" 7th edition, Elsevier Publishing, Amsterdam, 1966.	
8.6.5.2	Jungreis, Ervin.; "Spot Test Analysis, Clinical, Environmental, Forensic, and Geochemical Applications", Volume 75, John Wiley & Sons, 1985.	
8.6.5.3	1986-1987 Handbook and General Catalog, Pierce Chemical Company, Rockford, IL.	
8.6.5.4	Spots Tests, Systematic Analysis of Low Explosives, Bureau of Alcohol, Tobacco and Firearms, 6/88.	
8.7	Tear Gas and Pepper Sprays	
8.7.1	Purpose To identify tear gas products that may be encountered in casework: 2-chloroacetophenone (CN), o-chlorobenzylidenemalononitrile (CS) and oleoresin capsicum (OC), which contains the active component, capsaicin.	

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8.7.2	Safety Considerations	
8.7.2.1	Tear gas products are irritants, by definition, and will cause physical discomfort if inhaled. If working with spray products or highly concentrated clothing items, perform analysis in a fume hood.	
8.7.2.2	Avoid contact with skin and eyes.	
8.7.3	Minimum Standards and Controls	
8.7.3.1	Treat the questioned samples and any submitted control/standard samples in the same manner.	
8.7.3.2	When performing extractions also analyze a blank of the extraction liquid and an undisturbed portion of the substrate, if possible.	
8.7.3.3	When identifying a tear gas component, retention time data for samples and standards are needed from at least the polar or nonpolar GC columns along with retention time and spectral data from the GC/MS. Alternatively, an FTIR spectrum alone may be used for an identification, with comparison made to a standard spectrum, that was analyzed on the same instrument.	
8.7.4	Analytical Procedures	
8.7.4.1	Extraction of liquids (spray products)	
8.7.4.1.1	Weigh the canister to obtain the gross weight as received.	
8.7.4.1.2	CS and CN	
8.7.4.1.2.1	Oil based - Spray approximately 1 ml of the liquid into a vial and add about 1 ml of methanol. Analyze by GC and GCMS. Concentrate by evaporation or dilute with methanol as needed.	
8.7.4.1.2.2	Alcohol based - Spray approximately 5 ml, if available, into a small beaker and gently evaporate to dryness. The residue is taken up into a minimal volume of methanol for analysis by GC and GCMS. Alternatively, the dried residue can be analyzed by FTIR.	
8.7.4.1.3	Capsaicin	
8.7.4.1.3.1	Spray approximately 1 ml of the liquid into a vial and add about 1 ml of methanol. Analyze by GC and GCMS. Concentrate by evaporation or dilute with methanol as needed.	
8.7.4.2	Extraction of clothing items	
8.7.4.2.1	View the evidence item(s) under ultraviolet light or by using an alternative light source. Note any fluorescing areas, document as appropriate. If a known source canister is submitted also check the known liquid for fluorescence using ultraviolet light or an alternative light source.	
8.7.4.2.2	If a stained area is visible on the clothing, or becomes viewable by the process described above, remove a portion of it by cutting with scissors or a new scalpel. Remove an equivalent sized portion of an unstained area for use as a control, if necessary.	
8.7.4.2.3	If no stains are visible, remove portions of the garment from an area of the clothing where spray was reported to have been administered. Remove an equal sized portion of the	

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	clothing from an area of the garment farthest from the sampled area for use as a control, if necessary.	
8.7.4.2.4	Extract the cuttings with methanol, using the smallest volume to facilitate complete wetting of the cuttings and subsequent recovery of the methanol. Analyze by GC and GCMS. Concentrate sample or dilute with methanol as needed.	
8.7.5	Report Wording	
8.7.5.1	CS and CN:	
8.7.5.1.1	The Item _____ canister (was operational and) contained _____. _____, commonly known as _____, is an irritant.	
8.7.5.1.2	_____, commonly known as _____, was identified in the Item _____ extract.	
8.7.5.1.3	Report CS as o-chlorobenzylidenemalononitrile.	
8.7.5.1.4	Report CN as 2-chloroacetophenone.	
8.7.5.2	OC:	
8.7.5.2.1	The Item _____ canister (was operational and) contained _____. This is consistent with the labeling on the capsicum-based pepper spray container.	
8.7.5.2.2	Capsaicin (and dihydrocapsaicin) was (were) identified in the Item _____ extract. This (These) is a(are) component(s) of capsicum-based pepper sprays.	
8.7.5.3	If no CS, CN or OC are identified:	
8.7.5.3.1	No _____ were identified in the Item _____ extract.	
8.7.6	References	
8.7.6.1	Nowicki, J. "Analysis of Chemical Protection Sprays by GC/MS"; <i>Journal of Forensic Sciences</i> , 1982, 27, 3, 704-709	
8.7.6.2	Fung, T.; Jeffrey, W.; Beveridge, A.D. "The Identification of Capsaicinoids in Tear Gas Sprays"; <i>Journal of Forensic Sciences</i> , 1982, 27, 4, 812-821.	
8.7.6.3	Martz. "A Comparison of Ionization Techniques for Gas Chromatography/Mass Spectroscopy Analysis of Dye and Lachrymator Residues from Exploding Bank Security Devices"; <i>Journal of Forensic Sciences</i> , 1983, 28, 200.	
8.7.6.4	Ferslew. "Spectral Differentiation and Gas Chromatographic/Mass Spectrometric Analysis of the Lachrimators 2-chloroacetophenone and O-Chlorobenzylidene Malononitrile"; <i>Journal of Forensic Sciences</i> , 1986, 31, 658.	
8.7.6.5	Mongan, A.L.; Buel, E. "Identification of Dog Repellent in the Clothes of an Assault Suspect Using Gas Chromatography/Mass Spectrometry"; <i>Journal of Forensic Sciences</i> , 1995, 40, 3, 513-514.	
8.7.6.6	Gag, J.A.; Merck, N.F. "Concise Identifications of Commonly Encountered Tear Gases"; <i>Journal of Forensic Sciences</i> , 1977, 22, 22, 358-364.	
8.7.6.7	Illinois State Police, Trace Chemistry Procedures Manual, February 1997.	◆End